

IN THE SPECIFICATION:

Please amend paragraph number [0001] as follows:

[0001] This is a national stage application under 37 U.S.C. § 371 of PCT/US99/24013 filed on November 9, 1999, which claims the benefit of U.S. Provisional application Serial No. 60/108,455, filed November 12, 1998.

Please amend paragraph number [0004] as follows:

[0004] Conventional solid composite propellant binders utilize cross-linked elastomers in which prepolymers are cross-linked by chemical curing agents. As outlined in detail in U.S. Patent No. 4,361,526 to Allen, there are important disadvantages to using cross-linked elastomers as binders. Cross-linked elastomers must be cast within a short period of time after addition of the curative, which time period is known as the "pot life." Disposal of a cast, cross-linked propellant composition is difficult, and usually is accomplished by burning, which poses environmental problems. Furthermore, current state-of-the-art propellant compositions have serious problems that include their use of nonenergetic binders which have lower performance and high end-of-mix viscosities.

Please amend paragraph number [0005] as follows:

[0005] In view of the inherent disadvantages associated with the use of cross-linked elastomeric polymers as binder materials, there has been considerable interest in developing thermoplastic elastomers suitable as binders for solid, high energy compositions. However, many thermoplastic elastomers fail to meet important requirements expected of propellant formulations, particularly the requirement of being processible below about 120°C, it being desirable that a thermoplastic elastomeric polymer for use as a binder in a high energy system have a melting temperature of between about 60°C and about 120°C. The melting temperature is desirably at least about 60°C because the propellant composition may be subject to somewhat elevated temperatures during storage and transport, and significant softening of the propellant

composition at such elevated temperatures is unwanted. The setting of the melting temperature at not more than about 120°C is determined by the instability, at elevated temperatures, of many components which ordinarily go into high energy compositions, particularly oxidizer particulates and energetic plasticizers. Many thermoplastic elastomers exhibit high melt viscosities which preclude high solids loading and many show considerable creep and/or shrinkage after processing. Thermoplastic elastomers typically obtain their thermoplastic properties from segments that form glassy domains which may contribute to physical properties adverse to their use as binders. Cross-linkable thermoplastic elastomers are block copolymers with the property of forming physical cross-links at predetermined temperatures. One thermoplastic elastomer, e.g., ~~Kraton~~, Kraton™, brand TPE, obtains this property by having the glass transition point of one component block above room temperature. At temperatures below 109°C, the glassy blocks of ~~Kraton~~ Kraton™ form glassy domains and thus physically cross-link the amorphous segments. The strength of these elastomers depends upon the degree of phase separation. Thus, it remains desirable to have controlled, but significant, immiscibility between the two types of blocks, which is a function of their chemical structure and molecular weight. On the other hand, as the blocks become more immiscible, the melt viscosity increases, thus having a deleterious effect on the processibility of the material.

Please amend paragraph number [0007] as follows:

[0007] The preparation of energetic thermoplastic elastomers prepared from polyoxetane block copolymers has been proposed in U.S. Patent No. 4,483,978 to Manser ("the '978 Patent"), and U.S. Patent No. 4,806,613 to Wardle ("the '613 patent"), the complete disclosures of which are incorporated herein by reference to the extent that these disclosures are compatible with this invention. According to the latter, these materials overcome the disadvantages associated with conventional cross-linked elastomers such as limited pot-life, high end-of-mix viscosity, and scrap disposal problems.

Please amend paragraph number [0008] as follows:

[0008] The thermoplastic materials proposed by the '613 patent involve elastomers having both ~~(A) and (B)~~ A and B blocks, each derived from cyclic ethers, such as oxetane and oxetane derivatives and tetrahydrofuran ~~(THP)~~ ("THF") and ~~tetrahydrofuran~~ THF derivatives. The monomer or combination of monomers of the ~~(A)~~ A blocks are selected for providing a crystalline structure at usual ambient temperatures, such as below about 60°C, whereas the monomer or combination of monomers of the ~~(B)~~ B blocks are selected to ensure an amorphous structure at usual ambient temperatures, such as above about -20°C. Typical of these materials is the random block copolymer (poly(3-azidomethyl-3-methyloxetane)-poly(3,3-bis(azidomethyl)oxetane), also known as poly(AMMO/BAMO). These block copolymers have good energetic and mechanical properties. Additionally, the block copolymers can be processed without solvents to serve as binders in high performance, reduced vulnerability explosive, propellant, and gas generant formulations. Advantageously, the block copolymers exhibit good compatibility with most materials used in such energetic formulations.

Please amend paragraph number [0009] as follows:

[0009] However, the processing techniques disclosed in the '978 and '613 patents involve the use of halogenated solvents, such as methylene chloride. Several drawbacks have been associated with the use of the halogenated solvents disclosed in the '978 and '613 patents. One drawback is the detrimental impact that halogenated solvents have on the environment. Another drawback of halogenated solvents is attributable to the additional drying steps which the pre-polymer blocks are subject to after their formation. The pre-polymer blocks are typically dried either with chemical drying ~~agent~~, agents, e.g., desiccants followed by filtration or by the azeotropic removal of water. The azeotropic removal of water is performed with toluene, which is different from the solvent selected for linking the pre-polymer blocks. The performance of an additional drying step and the use of different solvents in the azeotropic drying step and the linking step complicate processing and increase overall processing time. Moreover, toluene does

not completely dissolve the end-capped blocks prior to the linking reaction and can interfere with the end-capping and linking catalysts. Yet another drawback associated with halogenated solvents is the relatively low concentrations of pre-polymer blocks and linking compounds that may be loaded in halogenated solvents for processing. The loading of the thermoplastic elastomer ingredients is limited by the solubility of the ingredients in the solvent. For example, the currently used process for forming thermoplastic elastomers by linking energetic polyether diols and diisocyanates typically use approximately 30-40% by weight solutions of the reactants in dichloromethane and 0.1% by weight tin catalyst. Additionally, completion of the reaction in halogenated solvents typically takes several days to a week.

Please amend paragraph number [0010] as follows:

[0010] United States Patent No. 4,393,199 to Manser ("the '199 Patent") describes the use of a non-halogenated solvent, nitromethane, during cationic polymerization of cyclic ethers. However, it has been found that cyclic ether pre-polymer blocks are not sufficiently soluble in nitromethane to adequately link the pre-polymer blocks once they are formed.

Please amend paragraph number [0012] as follows:

[0012] ~~It is, therefore, an object of this~~ The present invention to provide provides a method for the synthesis of energetic thermoplastic elastomers that addresses the aforementioned problems associated with the related art and realizes the advancement expressed above.

Please amend paragraph number [0013] as follows:

[0013] In accordance with the principles of this invention, ~~these and other objects~~ advantages are attained by a method of synthesizing an energetic thermoplastic elastomer binder that is in a solid state at room temperature and is derived from A blocks which are crystalline at temperatures below about 60°C and B blocks which are amorphous at temperatures above about -20°C. The A blocks may include one or more polyethers derived from monomers of oxetane

derivatives and/or tetrahydrofuran derivatives. The B blocks may include one or more polyethers derived from monomers of oxetane and its derivatives, tetrahydrofuran and its derivatives, and/or oxirane and its derivatives. The polyoxetane blocks A and polyoxirane blocks B may be linked by end-capping the blocks with diisocyanates and linking the end-capped blocks with difunctional linking chemicals in which each of the two terminal functional groups are reactive with an isocyanate moiety of the diisocyanate.

Please amend paragraph number [0014] as follows:

[0014] In accordance with one embodiment of this method, the A and B blocks are dissolved into solution comprising one or more non-halogenated solvents. The solvent or solvents selected preferably are capable of dissolving more than 25% by weight of the blocks (based on total weight of the solvents and blocks) into solution, more preferably at least 35% by weight into solution, and still more preferably 50% by weight into solution. The solvents preferably do not interfere with the ~~end-capping~~ end-capping catalyst, such as dibutyl tin dilaurate. Representative solvents include ethers, esters, and ketones. Tetrahydrofuran is the preferred solvent because of its excellent solubility. The A and B blocks are mixed together at approximately the stoichiometric ratios that the blocks are intended to be present in the energetic thermoplastic elastomer. The solution is then dried by azeotropic removal of water with excess solvent. The A and B blocks are end-capped in the solvent with one or more diisocyanates. The end-capping may be performed prior or subsequent to the mixing step, but subsequent to the drying step. The diisocyanate preferably has one isocyanate moiety which is more reactive, preferably at least about five times more reactive with the terminal hydroxyl group of each of the blocks than the other isocyanate moiety, whereby the more reactive isocyanate moiety tends to react with the terminal-hydroxyl groups of the blocks, leaving the less reactive isocyanate moiety free and unreactive. The mixture is reacted with a chain extender having two isocyanate-reactive groups that are sufficiently unhindered to react with the free and unreacted isocyanate groups of the end-capped blocks. In this manner, the end-capped blocks are linked, but not cross-linked, to form a thermoplastic elastomer.

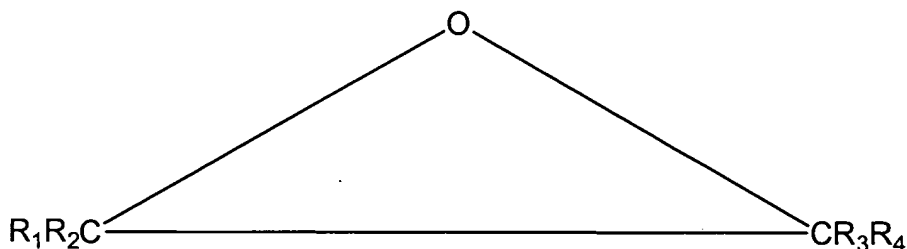
Please amend paragraph number [0015] as follows:

[0015] ~~It is still a further object of this invention to provide~~ The present invention also provides a method for making propellants, especially rocket propellants and gun propellants, explosives, gas generants, or the like containing an energetic thermoplastic elastomer binder by procedures including the above-discussed method.

Please amend paragraph number [0016] as follows:

[0016] ~~These and other objects, features, and advantages of the present invention will become apparent from the accompanying drawings and following detailed description which illustrate and explain, by way of example, the principles of this invention.~~

Please replace the formula appearing within paragraph number [0023] with the following structure (the lines on the lower left-hand corner incorrectly met):



Please amend paragraph number [0030] as follows:

[0030] The solids content of the high energy composition generally ranges from about 50 wt% to about 95 wt%, higher solids loading generally being preferred so long as such loading is consistent with structural integrity. The solids include fuel material particles and powders (collectively referred to herein as particulates), such as particulate aluminum, and/or oxidizer particulates. Representative fuels include aluminum, magnesium, boron, and beryllium. Representative oxidizers and co-oxidizers include ammonium perchlorate; hydroxylammonium

nitrate (HAN); ammonium dinitramide (ADN); hydrazinium nitroformate; ammonium nitrate; nitramines such as cyclotetramethylene tetranitramine (HMX) and cyclotrimethylene trinitramine (RDX), 2,4,6,8,10, 12-hexanitro-2,4,6,8,10,12-hexaazatetracyclo[5.5.0.^{05,9}.0^{3,11}]-dodecane or 2,4,6,8,10,12-hexanitrohexaazaisowurtzitane (CL-20 or HNIW), and/or 4,10-dinitro-2,6,8,12-tetraoxa-4,10-diazatetracyclo[5.5.0.0^{5,9}.0^{3,11}][5.5.0.0^{5,9}.0^{3,11}]-dodecane (TEX), and any combination thereof. In addition, the high energy composition may include minor amounts of additional components known in the art, such as bonding agents, burn rate modifiers, ballistic modifiers (e.g., lead), etc.

Please amend paragraph number [0033] as follows:

[0033] The oxetane homopolymer blocks may be formed according to the cationic polymerization technique taught by ~~Manser in U.S. the '199 Patent No. 4,393,199~~, the complete disclosure of which is incorporated herein by reference. The oxirane homopolymer blocks may be formed according to the technique taught by Willer et al. in U.S. Patent No. 5,120,827, the complete disclosure of which is incorporated herein by reference. The technique employs an adduct of a substance such as a diol, e.g., 1,4-butane diol (BDO), and a catalyst for cationic polymerization, e.g., BF₃-etherate. This adduct forms with the oxetane monomer an initiating species which undergoes chain extension until n moles of monomer have been incorporated in the molecule, n being the ratio of monomers to adduct present. By adjusting the ratio of monomers to adduct present, the average molecular weight of the polymer which forms may be adjusted. If two or more monomers are present, incorporation of the monomers will be generally random but may depend upon the relative reactivities of the monomers in the polymerization reaction.

Please amend paragraph number [0037] as follows:

[0037] Oxetane, THF, and oxirane polymer blocks normally have terminal isocyanate-reactive (e.g., hydroxyl) functions which are end-capped with the diisocyanates in

accordance with the invention. Preferably, a first one of the isocyanate moieties of the end-capping compound is substantially more reactive with the terminal-hydroxyl moieties of the polymer blocks than the other (e.g., second) isocyanate moiety. One of the problems with linking these types of polymer blocks is that substituted oxetane-derived hydroxyl end groups units have neopentyl structures, whereby the terminal primary hydroxyl moieties are substantially hindered and therefore less reactive. The blocks derived from the oxirane derivatives are secondary alcohols, making their hydroxyl groups less reactive than the primary hydroxyl group of the oxetane-derived ~~A block.~~ A block. The diisocyanate preferably is selected so that one of the isocyanate groups is capable of reacting with a hydroxyl-group of the polymer blocks while the other isocyanate moiety remains free and unreacted. Diisocyanates are preferably used because isocyanates of higher functionality would result in undesirable levels of cross-linking. The different reactivities of the isocyanate moieties are desirable to ensure that substantial chain extension through linking of like blocks does not occur. Thus, for purposes of this invention, one isocyanate moiety of the diisocyanate should preferably be approximately five times more reactive with terminal hydroxyl groups of oxetane and oxirane blocks than the other group. Preferably one isocyanate moiety is at least about ten times more reactive than the other.

Please amend paragraph number [0040] as follows:

[0040] In one variant embodiment, the A blocks and B blocks are reacted separately with the diisocyanate, so that there is no competition of the blocks for diisocyanate molecules and each separate end-capping reaction may be carried to substantial completion. The diisocyanate may react more rapidly with one block than the other, but this difference can be compensated for by a longer reaction time with the slower reacting block. The reactivity of the terminal hydroxyl groups varies according to steric factors and also according to side-chain moieties. Energetic oxetanes, for example, generally have side-chain moieties that are electron-withdrawing, making their terminal hydroxyl groups less reactive. Once end-capped with diisocyanate, the reactivities of the polymers for linking purposes are essentially dependent

only upon the reactivity of the free isocyanate, not on the chemical makeup of the polymer chain itself. Thus end-capped-(A)- A blocks are substantially as reactive as end-capped-(B)- B blocks.

Please amend paragraph number [0041] as follows:

[0041] The end-capping reaction is promoted by a suitable urethane catalyst. A preferred class of catalysts ~~include~~ includes organic tin compounds with at least one and preferably two labile groups, such as chloride or acetate, bound directly to the tin. Suitable tin catalysts include diphenyl tin dichloride, dibutyl tin dichloride, dibutyl tin dilaurate, dibutyl tin diacetate. Tertiary amine catalysts may also be used.

Please amend paragraph number [0048] as follows:

[0048] The end-capping reaction and linking reaction are carried out in a suitable non-halogenated solvent, e.g., one which dissolves the polymer and does not react with the free isocyanate moieties. Although insubstantial amounts of halogenated solvent may be present, the solution is preferably completely free of any halogenated solvent. The non-halogenated solvent should not react in the urethane reaction and forms an azeotrope with water. Suitable dry solvents include cyclic ethers such as ~~tetrahydrofuran (THF)~~ THF and 1,4-dioxane; non-cyclic ethers such as ethylene glycol dimethyl ether; ketones such as methyl ethyl ketone ("MEK"); and esters such as ethyl acetate. Of these, THF is preferred because of its excellent solubility characteristics.

Please amend paragraph number [0050] as follows:

[0050] In the end-capping and block linking steps, the reaction can be followed with ~~NMR- numclear maghnetic resonance ("NMR") and IR- infrared ("IR") spectroscopy.~~ NMR- nuclear magnetic resonance ("NMR") and IR- infrared ("IR") spectroscopy. With NMR, the urethane-forming reaction can be followed through the methylene groups on the polymer adjacent to the terminal hydroxyl groups. With IR, the change from isocyanate to urethane can be directly followed.

Please amend paragraph number [0059] as follows:

[0059] Under an argon atmosphere, 14.94 grams of butane diol was added to a flame dried 5 liter round-bottomed flask charged with ~~1.340~~ 1340 ml of dry methylene chloride. To this mixture, 11.77 grams of borontrifluoride-etherate was added and the reaction was allowed to proceed for one hour at room temperature. The reactor was then cooled to -10°C and 937.78 grams of the 3-azidomethyl-3-methyloxetane was added. The solution was allowed to come to room temperature and left to react for three days. The reaction was then quenched by the addition of 50 ml of saturated brine solution. The organic phase was separated off and washed with 100 ml of 10 wt% sodium bicarbonate solution before the solvent was removed on a rotovapor. The resulting liquid was then poured into 5 liters of methanol to precipitate the polymer, which was filtered from the solution and dried under vacuum at 30°C.

Please amend paragraph number [0060] as follows:

[0060] In a one liter round bottom flask, 65 grams of dry difunctional poly(3-azidomethyl-3-methyloxetane) with a hydroxyl equivalent weight of 3121 and 35 grams of dry poly(3,3-bis(azidomethyl)oxetane) with a hydroxyl equivalent weight of 3235 were dissolved in 650 grams of dry tetrahydrofuran. The solution was concentrated and dried by evaporation of the tetrahydrofuran under reduced pressure via a rotovapor until 100 grams of the solvent remained. To this solution, ~~0.1-grams~~ gram of diphenyltin dichloride and 5.41 grams of toluene-2,4-diisocyanate were added while stirring with a magnetic stirrer at ambient temperature and pressure. After 24 hours, 1.40 grams of butane-1,4-diol was added causing the solution to become steadily more viscous. After another 24 hours, the solution was poured into methanol in a volume ratio of 1:5. The methanol was decanted off, and the precipitated polymer was washed three times with fresh methanol (1:5 volume ratio) to give a rubbery granular product with the properties set forth below:

$$M_n = 20480$$

$$M_w = 120800$$

$$M_w/M_n = 5.9$$

$$E^{1.0} (\text{psi}) = 754$$

$$\epsilon_m (\%) = 204$$

$$\epsilon_f (\text{failure}) (\%) = 229$$

$$\sigma_m (\text{psi}) = 180$$

$$\sigma_m (\text{corrected}) (\text{psi}) = 558$$

$$\text{ShoreA} = 64$$

Please amend paragraph number [0061] as follows:

[0061] In a 100 ml round bottom flask, 6.5 grams of dry difunctional poly(3-azidomethyl-3-methyloxetane) with a hydroxyl equivalent weight of 3040 and 3.5 grams of dry poly(3,3-bis(azidomethyl)oxetane) with a hydroxyl equivalent weight of 3235 were dissolved in 60 ml of dry tetrahydrofuran. The solution was concentrated and dried by evaporation of the tetrahydrofuran under reduced pressure via a rotovapor until 10 grams of the solvent remained. To this solution, 0.25 ml of dibutyltin dilaurate and 0.561-grams-gram of toluene-2,4-diisocyanate were added while stirring with a magnetic stirrer at ambient temperature and pressure. After 15 minutes, 0.145-grams-gram of butane-1,4-diol was added causing the solution to become steadily more viscous. After another 30 minutes, the solution was poured into methanol in a volume ratio of 1:5. The methanol was decanted off, and the precipitated polymer was washed three times with fresh methanol (1:5 volume ratio) to give a rubbery granular product with the properties set forth below:

$$M_n = 15404$$

$$M_w = 67277$$

$$M_w/M_n = 4.37$$

Please amend paragraph number [0062] as follows:

[0062] In a 100 ml round bottom flask, 6.5 grams of dry difunctional poly(3-azidomethyl-3-methyloxetane) with a hydroxyl equivalent weight of 3040 and 3.5 grams of dry poly(3,3-bis(azidomethyl)oxetane) with a hydroxyl equivalent weight of 3235 were dissolved in 60 ml of dry 1,4-dioxane. The solution was concentrated and dried by evaporation of the 1,4-dioxane under reduced pressure via a rotovapor until 12 grams of the solvent remained. To this solution, 0.3 ml of dibutyltin dilaurate and 0.561 ~~grams~~ gram of toluene-2,4-diisocyanate were added while stirring with a magnetic stirrer at ambient temperature and pressure. After one hour, 0.145 ~~grams~~ gram of butane-1,4-diol was added causing the solution to become steadily more viscous. After another two hours, the solution was poured into methanol in a volume ratio of 1:5. The methanol was decanted off, and the precipitated polymer was washed three times with fresh methanol (1:5 volume ratio) to give a rubbery granular product with the properties set forth below:

$$M_n = 21230$$

$$M_w = 116272$$

$$M_w/M_n = 5.48$$

Please amend paragraph number [0063] as follows:

[0063] In a 500 ml round bottom flask, 18 grams of dry difunctional poly(3-azidomethyl-3-methyloxetane) with a hydroxyl equivalent weight of 3356 and 6 grams of dry poly(3,3-bis(azidomethyl)oxetane) with a hydroxyl equivalent weight of 3235 were dissolved in 200 ml of dry ethyl acetate. The solution was concentrated and dried by evaporation of the ethyl acetate under reduced pressure via a rotovapor until 34 grams of the solvent remained. To this solution at 40°C, 0.024 ~~grams~~ gram of diphenyltin dichloride and 1.26 grams of toluene-2,4-diisocyanate were added while stirring with a magnetic stirrer. After one hour, 0.33 ~~grams~~ gram of butane-1,4-diol was added causing the solution to become steadily more viscous. After another 48 hours, the solution was poured into methanol in a volume ratio of 1:5. The

methanol was decanted off, and the precipitated polymer was washed three times with fresh methanol (1:5 volume ratio) to give a rubbery granular product with the properties set forth below:

$$M_n = 13880$$

$$M_w = 43310$$

$$M_w/M_n = 3.12$$

Please amend paragraph number [0064] as follows:

[0064] In a 100 ml round bottom flask, 6.5 grams of dry difunctional poly(3-azidomethyl-3-methyloxetane) with a hydroxyl equivalent weight of 3040 and 3.5 grams of dry poly(3,3-bis(azidomethyl)oxetane) with a hydroxyl equivalent weight of 3235 were dissolved in 70 ml of dry tetrahydrofuran. The solution was concentrated and dried by evaporation of the tetrahydrofuran under reduced pressure via a rotovapor at 50°C until 5 grams of the solvent remained. To this solution at 50°C, 0.5 ml of dibutyltin dilaurate and 0.561-grams ~~gram~~ of toluene-2,4-diisocyanate were added while stirring with a magnetic stirrer. After three minutes, 0.145-grams ~~gram~~ of butane-1,4-diol was added causing the solution to become steadily more viscous. After another five minutes, the solution was diluted with 20 ml of tetrahydrofuran and 1 ml of methanol. This solution was then precipitated in methanol in a volume ratio of 1:5. The methanol was decanted off, and the precipitated polymer was washed three times with fresh methanol (1:5 volume ratio) to give a rubbery granular product with the properties set forth below:

$$M_n = 17350$$

$$M_w = 103231$$

$$M_w/M_n = 5.95$$

Please amend paragraph number [0067] as follows:

[0067] Molecular weight distribution was determined by gel permeation chromatography using polystyrene standards, with the results shown in FIG. 1. The ~~GPC gel permeation chromatography~~ (“GPC”) trace in FIG. 1 demonstrates that the prepolymers were linked to produce a copolymer having a higher molecular weight and dispersivity than the homopolymer blocks. The ~~DMA dynamic mechanical analysis~~ (“DMA”) trace in FIG. 2 shows the melt transition of random block (BAMO-GAP)_n at 75-80°C with a material modulus reducing only slowly before this point.

Please amend paragraph number [0068] as follows:

[0068] In a 500 ml round bottom flask, 45 grams of difunctional poly(3-azidomethyl-3-methyloxetane) with a hydroxyl equivalent weight of 3125 and 15 grams of poly(3,3-bis(azidomethyl)oxetane) with a hydroxyl equivalent weight of 3152 were dissolved in 300 ml of tetrahydrofuran. The solution was concentrated and dried by evaporation of the tetrahydrofuran or a rotovapor until a solution with 90 grams of solvent remained. To this ~~solution~~ solution 0.6 ~~grams~~ gram of dibutyltin dichloride and 3.34 grams of toluene-2,4-diisocyanate ~~was~~ were added, and the mixture was allowed to react for 3 hours to end-cap the prepolymer.

Please amend paragraph number [0069] as follows:

[0069] For Example 9, 0.22 ~~grams~~ gram of butanediol was added to one quarter of the isocyanate end-capped prepolymer mixture. The reaction was allowed to continue for 14 hours before it was precipitated with methanol in a volume ratio of 1:5. The methanol was decanted off, and the precipitated polymer was washed three times with fresh methanol (1:5 volume ratio) to give a rubbery granular product.

Please amend paragraph number [0070] as follows:

[0070] For Example 10, a urethane oligomer was derived from a mixture of 2 ml of tetrahydrofuran, 0.42-~~grams~~ gram of toluene-2,4-diisocyanate, 0.43-~~grams~~ gram of butane-1,4-diol, and 0.1-~~grams~~ gram of dibutyltin dichloride, which were allowed to react for one hour. The urethane oligomer was then added to one quarter of the isocyanate end-capped prepolymer mixture and allowed to react for 14 hours before it was precipitated with methanol in a volume ratio of 1:5. The methanol was decanted off, and the precipitated polymer was washed three times with fresh methanol (1:5 volume ratio) to give a rubbery granular product.

Please amend paragraph number [0071] as follows:

[0071] For Example 11, a urethane oligomer was derived from a mixture of 2 ml of tetrahydrofuran, 0.83-~~grams~~ gram of toluene-2,4-diisocyanate, 0.65-~~grams~~ gram of butane-1,4-diol, and 0.1-~~grams~~ gram of dibutyltin dichloride, which were allowed to react for one hour. The urethane oligomer was then added to one quarter of the isocyanate end-capped prepolymer mixture and allowed to react for 14 hours before it was precipitated with methanol in a volume ratio of 1:5. The methanol was decanted off, and the precipitated polymer was washed three times with fresh methanol (1:5 volume ratio) to give a rubbery granular product.